NELAC Quality Systems
Appendix D – Essential Quality Control Requirements

> NOTE: THIS DRAFT APPENDIX ON QUALITY ASSURANCE FOR ASBESTOS ANALYSIS INCLUDES DRAFT SECTIONS FOR ANALYSIS OF WATER, WASTEWATER AND AIR BY TEM. SECTIONS ON ANALYSIS OF BULK SAMPLES BY TEM, THE ANALYSIS OF WORKPLACE AIR BY PCM, AND THE ANALYSIS OF BULK SAMPLES BY PLM ARE BEING DEVELOPED. COMMENTS ON THE DRAFT ARE WELCOME. PLEASE FORWARD ALL COMMENTS TO MIKE BEARD AT mebeard@rti.org.

D.7 ASBESTOS TESTING

These standards apply to laboratories undertaking the examination of asbestos samples. These standards are organized by analytical technique including transmission electron microscopy (TEM) for the analysis of water, wastewater, air, and bulk samples; phase contrast microscopy (PCM) for analysis of workplace air; and polarized light microscopy (PLM) for analysis of bulk samples. These procedures for asbestos analysis involve sample preparation followed by detection of asbestos.

D.7.1 Negative Controls

D.7.1.1　　TEM

D.7.1.1.1　　Water and Wastewater

a) Blank determinations shall be made prior to sample collection. When using polyethylene bottles, one bottle from each batch, or a minimum of one from each 24 shall be tested for background level. When using glass bottles, four bottles from each 24 shall be tested. An acceptable bottle blank level is defined as < 0.01 MFL > 10 μm. (Method 100.2, Section 8.2)

b) A process blank sample consisting of fiber-free water shall be run before the first field sample. The quantity of water shall be $\geq$ 10 mL for 25-mm diameter filter and $\geq$ 50 mL for a 47-mm diameter filter. (Method 100.2, Section 11.8)

D.7.1.1.2　　Air

a) A blank filter shall be prepared with each set of samples. A blank filter shall be left uncovered during preparation of the sample set and a wedge from that blank filter shall be prepared alongside wedges from the sample filters. At minimum, the blank filter shall be analyzed for each 20 samples analyzed. (40 CFR Part 763, Appendix A to Subpart E (AHERA), Table 1)

b) Maximum contamination on a single blank filter shall be no more than 53 structures/mm^2. Maximum average contamination for all blank filters shall be no more than 18 structures/mm^2. (AHERA, III.F.2)

D.7.1.1.3   Bulk

D.7.1.2   PCM

D.7.1.3   PLM

D.7.2 Test Variability/Reproducibility

D.7.2.1   TEM

Quality assurance analyses shall be performed regularly covering all time periods, instruments, tasks, and personnel. The selection of samples shall be random and samples of special interest may be included in the selection of samples for quality assurance analyses. When possible, the checks on personnel performance shall be executed without their prior knowledge. A disproportionate number of analyses shall not be performed prior to internal or external audits.

D.7.2.1.1   Water and Wastewater

All analyses must be performed on relocator grids so that other laboratories can easily repeat analyses on the same grid openings. Quality assurance analyses shall not be postponed during periods of heavy workloads. The total number of QA samples and blanks must be greater than or equal to 10% of the total sample workload. Precision of analyses is related to concentration, as gleaned from interlaboratory proficiency testing. Relative standard deviations (RSD) for amphibole asbestos decreased from 50% at 0.8 MFL to 25% at 7 MFL in interlaboratory proficiency testing, while RSD for chrysotile was higher, 50% at 6 MFL.

a) Replicate – A second, independent analysis shall be performed on the same grids but on different grid openings than used in the original analysis of a sample. Results shall be within 1.5x of Poisson standard deviation. This shall be performed at a frequency of 1 per 100 samples. (Method 100.2, Table 2)

b) Duplicate – A second aliquot of sample shall be filtered through a second filter, prepared and analyzed in the same manner as the original preparation of that sample. Results shall be within 2.0x of Poisson standard deviation. This shall be performed at a frequency of 1 per 100 samples. (Method 100.2, Table 2)

c) Verified Analyses – A second, independent analysis shall be performed on the same grids and grid openings used in the original analysis of a sample. The two sets of results shall be compared according to Turner and Steel (NISTIR 5351). This shall be performed at a frequency of 1 per 20 samples. Qualified analysts

must maintain an average of 80% true positives, $\leq$ 20% false negatives, and $\leq$ 10% false positives.

D.7.2.1.2   Air

All analyses must be performed on relocator grids so that other laboratories can easily repeat analyses on the same grid openings.   *(T. Davis to provide language on air reproducibility.)*

a) Replicate – A second, independent analysis shall be performed in accordance with Section D.7.2.1.1.a. (AHERA, Table III)

b) Duplicate – A second wedge from a sample filter shall be prepared and analyzed in the same manner as the original preparation of that sample. Results shall be within 2.0x of Poisson standard deviation. This shall be performed at a frequency of 1 per 100 samples. (AHERA, Table III)

c) Verified Analyses – A second, independent analysis shall be performed on the same grids and grid openings in accordance with Section D.7.2.1.1.c. (AHERA, Table III)

D.7.2.1.3   Bulk

D.7.2.2   PCM

D.7.2.3   PLM

D.7.3 Other Quality Control Measures

D.7.3.1   TEM

D.7.3.1.1   Water and Wastewater

a) Filter preparations shall be made from all six asbestos types from NIST SRMs 1866 and 1867. These preparations shall have concentrations between 1 and 20 structures (> 10μm) per 0.01 mm^2. One of these preparations shall be analyzed independently at a frequency of 1 per 100 samples analyzed. Results shall be evaluated as verified asbestos analysis in accordance with Turner and Steel (NISTIR 5351).

b) NIST SRM 1876b must be analyzed annually by each analyst. Results shall be evaluated in accordance with limits published for that SRM. Comment: This SRM is not strictly appropriate for waterborne asbestos but analysts can demonstrate general TEM asbestos competence by producing results within the published limits of this (the only recognized TEM counting standard) SRM.

D.7.3.1.2   Air

a) Filter preparations shall be made from all six asbestos types in accordance with Section D.7.3.1.1.a.

b) NIST SRM 1876b must be analyzed annually in accordance with Section D.7.3.1.1.b.

D.7.3.1.3   Bulk

D.7.3.2   PCM

D.7.3.3   PLM

D.7.4 Method Evaluation

D.7.4.1   TEM

D.7.4.1.1   Water, Wastewater and Air

In order to ensure the accuracy of reported results, the following procedures shall be in place:

a) Demonstration of Capability – (Refer to Section 5.10.2.1) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, or method.

b) Proficiency Test Samples – (Refer to Section 5.4.2j or 5.5.3.4) The results of such analysis shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.7.4.1.2   Bulk

D.7.4.2   PCM

D.7.4.3   PLM

D.7.5 Asbestos Measurement System Calibration

D.7.5.1   TEM

D.7.5.1.1   Water and Wastewater

All calibrations listed below (unless otherwise noted) must be performed under the same analytical conditions used for routine asbestos analysis and must be recorded in a notebook and include date and analyst's signature. Frequencies stated below may be reduced to "before next use" if no samples are analyzed after the last calibration period has expired. Likewise, frequencies may have to be increased following non-routine maintenance or unacceptable calibration performance. All

calibration data must be displayed on control charts that show trends over time.

a) Magnification Calibration – Magnification calibration must be done at the fluorescent screen, with the calibration specimen at the eucentric position, at the magnification used for fiber counting, generally 10,000 and 20,000x. A logbook must be maintained with the dates of the calibration recorded. Calibrations shall be performed monthly to establish the stability of magnification. (Method 100.2, Section 10.1)

b) Camera Constant – The camera length of the TEM in the electron diffraction (ED) mode must be calibrated before ED patterns of unknown samples are observed. The diffraction specimen must be at the eucentric position for this calibration. This calibration shall allow accurate (< 10% variation) measurement of layer-line spacings on the medium used for routine measurement, i.e., the phosphor screen or camera film. This must also allow accurate (< 5% variation) measurement of zone axis ED patterns on permanent media, e.g., film. Calibrations shall be performed monthly to establish the stability of the camera constant. (Method 100.2, Section 10.1)

c) Spot Size – The diameter of the smallest beam spot at crossover must be less than 250 nm as calibrated quarterly. (Method 100.2, Section 10.3)

d) Beam Dose - The beam dose shall be calibrated so that beam damage to chrysotile is minimized, specifically so that an electron diffraction pattern from a single fibril ≥ 1 μm in length from a NIST SRM chrysotile sample is stable in the electron beam dose for at least 15 seconds.

e) EDXA System

1) The x-ray energy vs. channel number for the EDXA system shall be calibrated to within 20 eV for at least two peaks between 0.7 keV and 10 keV. One peak shall be from the low end (0.7 keV to 2 keV) and the other peak from the high end (7 keV to 10 keV) of this range. The calibration of the x-ray energy shall be checked prior to each analysis of samples and recalibrated if out of the specified range.

2) The ability of the system to resolve the Na Kα line from the Cu L line shall be confirmed quarterly by obtaining a spectrum from the NIST SRM 1866 crocidolite sample on a copper grid.

3) The k-factors relative to Si shall be calibrated semiannually for Mg, Si, Ca, and Fe using NIST SRM 2063. The k-factor for Mg/Fe must be 1.5 or less (Section 10.4, Method 100.2). Additionally, k-factors from Na and Al must be checked at the same frequency

using suitable materials such as albite, kaersutite, or SRM 99a. The Na/Si k-factor must be 4.0 or less.

4) The detector resolution shall be checked quarterly to ensure a full-width half-maximum resolution of < 175 eV at Mn Kα (5.90 keV).

5) The relative sensitivity (k-factors) factors relative to Si for elements found in asbestos (Na, Mg, Al, Si, Ca, Fe) shall be determined so that:

    i.   the k-factors shall be determined to a precision (2s) within 10% relative to the mean value obtained for Mg, Al, Si, Fe, and within 20% relative to the mean value obtained for Na;

    ii.   the k-factor relative to Si for Na shall be between 1.0 and 4.0, for Mg and Fe shall be between 1.0 and 2.0, and for Al and Ca shall be between 1.0 and 1.75; and

    iii.   the k-factor for Mg relative to Fe on SRM 2063(a) or other standard traceable to NIST shall be 1.5 or less.

6) The portions of a grid in a specimen holder for which abnormal x-ray spectra are generated under routine asbestos analysis conditions shall be determined and these areas shall be avoided in asbestos analysis.

7) The sensitivity of the detector for collecting x-rays from small volumes shall be documented quarterly by collecting resolvable Mg and Si peaks from a unit fibril of NIST SRM 1866 chrysotile.

f) Low Temperature Asher - The low temperature asher shall be calibrated quarterly by determining a calibration curve for the weight vs. ashing time of collapsed mixed-cellulose-ester (MCE) filters.

g) Grid Openings - The magnification of the grid opening measurement system shall be calibrated using an appropriate standard at a frequency of 20 openings/20 grids/lot of 1000 or 1 opening/sample. The variation in the calibration measurements (2s) is <5% of the mean calibration value.

D.7.5.1.2   Air

All calibrations must be performed in accordance with Section D.7.5.1.1, with the exception of magnification. Magnification calibration must be done at the fluorescent screen, with the calibration specimen at the eucentric position, at the magnification used for fiber counting, generally 15,000 to 20,000x (AHERA, III.G.1.c). A logbook must be maintained with the dates of the calibration recorded. Calibrations shall be performed monthly to establish the stability of magnification.

D.7.5.1.3   Bulk

D.7.5.2   PCM

D.7.5.3   PLM

D.7.6 Analytical Sensitivity

D.7.6.1   TEM

D.7.6.1.1   Water and Wastewater

An analytical sensitivity of 200,000 fibers per liter (0.2 MFL) is required for each sample analyzed. Analytical sensitivity is defined as the waterborne concentration represented by the finding of one asbestos structure in the total area of filter examined. This value will depend on the fraction of the filter sampled and the dilution factor (if applicable).

D.7.6.1.2   Air

An analytical sensitivity of 0.005 structures/mm^2 is required for each sample analyzed. Analytical sensitivity is defined as the airborne concentration represented by the finding of one asbestos structure in the total area of filter examined. This value will depend on the effective surface area of the filter, the filter area analyzed, and the volume of air sampled (AHERA, Table I).

D.7.6.1.3   Bulk

D.7.6.2   PCM

D.7.6.3   PLM

D.7.7 Data Reduction

D.7.7.1   TEM

D.7.7.1.1   Water and Wastewater

a) The concentration of asbestos in a given sample must be calculated in accordance with Method 100.2, Section 12.1. Refer to Section 5.10.6, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Measurement Uncertainties – The laboratory must calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample. *(Note to Asbestos Subcommittee: Need expansion of this section pending NELAC committee decision.)*

D.7.7.1.2   Air

a) The concentration of asbestos in a given sample must be calculated in accordance with the method utilized, e.g., AHERA. Refer to Section 5.10.6, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) *Measurement Uncertainties – The laboratory must calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample. (Note to Asbestos Subcommittee: Need expansion of this section pending NELAC committee decision.)*

D.7.7.1.3   Bulk

D.7.7.2   PCM

D.7.7.3   PLM

D.7.8 Quality of Standards and Reagents

D.7.8.1   TEM

D.7.8.1.1   Water, Wastewater and Air

a) The quality control program shall establish and maintain provisions for asbestos standards.

1) Reference standards that are used in an asbestos laboratory shall be obtained from the National Institute of Standards and Technology (NIST), EPA, or suppliers who participate in supplying NIST standards or NIST traceable asbestos. Any reference standards purchased outside the United States shall be traceable back to each country's national standards laboratory. Commercial suppliers of reference standards shall conform to ANSI N42.22 to assure the quality of their products.

2) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22-1995, Section 8, Certificates.

b) All reagents used shall be analytical reagent grade or better.

c) The laboratory shall have mineral fibers or data from mineral fibers that will allow differentiating asbestos from at least the following "look-alikes": fibrous talc, sepiolite, wollastonite, attapulgite (palygorskite), halloysite, vermiculite scrolls, antigorite, lizardite, pyroxenes, hornblende, richterite, winchite, or any other asbestiform minerals that are suspected as being present in the sample. *(Reference document that is to be provided by Dan Crane)*.

D.7.8.1.2   Bulk

D.7.8.2   PCM

D.7.8.3   PLM

D.7.9   Constant and Consistent Test Conditions

D.7.9.1   TEM

D.7.9.1.1   Water, Wastewater and Air

a) The laboratory shall establish and adhere to written procedures to minimize the possibility of cross-contamination between samples.

b) Carbon-Coating Filter Segments – Coating must be performed with a high-vacuum evaporation unit equipped with a rotating tilting stage. The carbon rods should be sharpened by a carbon rod sharpener to necks of about 4 mm long and 1 mm in diameter. The rods should be installed in the evaporator in such a manner that the points are approximately 10 cm from the surface of the filter. (Method 100.2, Section 11.18.1; AHERA, III.F.7.e)

D.7.9.1.2   Bulk

D.7.9.2   PCM

D.7.9.3   PLM